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Vacuum technology — Vacuum gauges — Characterization of quadrupole mass spectrometers for partial pressure measurement

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National foreword

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Foreword

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This document was prepared by Technical Committee ISO/TC 112, *Vacuum technology*.

Introduction

Quadrupole mass spectrometers (QMSs) are nowadays used not only in vacuum technology for leak detection and residual gas analysis but also in the process industry as an instrument to provide quantitative analysis in processes and to control processes such as physical and chemical vapour deposition, and etch processes. They are also used for quantitative outgassing rate measurements which are important to characterize vacuum components for critical applications like in the EUV lithography, semiconductor industry or medical instruments.

Total pressure, composition of the gas mixture, settings and the operational history of QMSs, to name a few, have a significant influence on the measured signal, its uncertainty and interpretation. For this reason, it is not possible to calibrate QMS for all its possible applications. Instead, it has either to be calibrated for the special conditions at use or for a standardized condition. It is the purpose of this document to establish such conditions.

There is also a need for standardization in order to enable the users of QMSs to compare the devices of different manufactures and to use the QMS properly.

This document provides standardized calibration procedures for QMSs for some important applications. These have been selected from the results of a survey of the international project EMRP (European Metrological Research Programme) IND12 which was conducted in 2013. This survey included manufacturers, distributors and users of quadrupole mass spectrometers.

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1 Scope

This document describes procedures to characterize quadrupole mass spectrometers (QMSs) with an ion source of electron impact ionization and which are designed for the measurement of atomic mass-to-charge ratios $m/z < 300$.

This document is not applicable to QMSs with other ion sources, such as chemical ionization, photo-ionization or field ionization sources and for the measurements of higher *m/z*, which are mainly used to specify organic materials.

It is well known from published investigations on the metrological characteristics of quadrupole mass spectrometers that their indications of partial pressures depend significantly on the settings of the instrument, the total pressure, and the composition of the gas mixture. For this reason, it is not possible to calibrate a quadrupole mass spectrometer for all possible kinds of use. The characterization procedures described in this document cover the applications of continuous leak monitoring of a vacuum system, leak rate measurement with tracer gas, residual gas analysis and outgassing rate measurements. The user can select that characterization procedure that best suits his or her needs. These characterization procedures can also be useful for other applications.

It is also well known that the stability of several parameters of quadrupole mass spectrometers, in particular sensitivity, are rather poor. Therefore, when a parameter has been calibrated, it needs frequent recalibration when accuracy is required. For practical reasons this can only be accomplished by *in situ* calibrations. To this end, this document not only describes how a quadrupole mass spectrometer can be calibrated by a calibration laboratory or a National Metrological Institute with direct traceability to the System International (SI), but also how calibrated parameters can be frequently checked and maintained *in situ*.

By their physical principle, quadrupole mass spectrometers need high vacuum within the instrument. By reducing dimensions or by special ion sources combined with differential pumping the operational range can be extended to higher pressures, up to atmospheric pressure. This document, however, does not include quadrupole mass spectrometers with differential pumping technology. Therefore, it does not cover pressures exceeding 1 Pa on the inlet flange of the quadrupole mass spectrometer.

This document does not describe how the initial adjustment of a quadrupole mass spectrometer by the manufacturer or by a service given order by the manufacturer should be made. The purpose of such an initial adjustment is mainly to provide a correct *m/z* scale, constant mass resolution or constant transmission, and is very specific to the instrument. Instead, it is assumed for this document that a manufacturer's readjustment procedure exists which can be carried on-site by a user. This procedure is intended to ensure that the quadrupole mass spectrometer is in a well-defined condition for the characterization.

It is the intention of this document that the user gets the best possible metrological quality from his quadrupole mass spectrometer. From investigations it is known that in most cases this can be achieved in the so called "scan mode". The bar graph may also be of an adequate quality depending on the software used for evaluation of the data taken by the quadrupole mass spectrometer. The trend mode, however, often involves the additional uncertainty that a shift of the peak value position on the mass scale causes a shift in ion current. For this reason, the scan mode is preferable for most of the measurement procedures of this document.